

FORM PTO-1399  
(REV. 11-2000)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

Mo-6342/WW-5522

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)

To Be Assigned

097856545

INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/EP99/08779

November 15, 1999

November 17, 1998

TITLE OF INVENTION Method For The Production of Low-Viscous Water-Soluble Cellulose Ethers

APPLICANT(S) FOR DO/EO/US SCHLESIGER, Hartwig

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND or SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ has been communicated by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
  - a. ☐ is attached hereto.
  - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ have been communicated by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

## Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND or SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

Abstract  
Form PTO 1449

21. ☒ The following fees are submitted:**BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):**Neither international preliminary examination fee (37 CFR 1.482)  
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO  
and International Search Report not prepared by the EPO or JPO ..... \$1000.00International preliminary examination fee (37 CFR 1.482) not paid to  
USPTO but International Search Report prepared by the EPO or JPO ..... \$860.00International preliminary examination fee (37 CFR 1.482) not paid to USPTO  
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$710.00International preliminary examination fee (37 CFR 1.482) paid to USPTO  
but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$690.00International preliminary examination fee (37 CFR 1.482) paid to USPTO  
and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00**ENTER APPROPRIATE BASIC FEE AMOUNT =****CALCULATIONS PTO USE ONLY**Surcharge of \$130.00 for furnishing the oath or declaration later than \_\_\_\_\_ months from the earliest claimed priority date (37 CFR 1.492(e)). ☐ 20 ☐ 30

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	13 -20 =	0	x \$18.00
Independent claims	1 -3 =	0	x \$80.00

MULTIPLE DEPENDENT CLAIM(S) (if applicable) + \$270.00

**TOTAL OF ABOVE CALCULATIONS =**Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above  
are reduced by 1/2.**SUBTOTAL =**Processing fee of \$130.00 for furnishing the English translation later than \_\_\_\_\_ months from the earliest claimed priority date (37 CFR 1.492(f)). ☐ 20 ☐ 30**TOTAL NATIONAL FEE =**Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be  
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +**TOTAL FEES ENCLOSED =**Amount to be  
refunded: \$

charged: \$

- a. ☐ A check in the amount of \$ \_\_\_\_\_ to cover the above fees is enclosed.
- b. ☒ Please charge my Deposit Account No. 13-3848 in the amount of \$ 900.00 to cover the above fees.  
A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any  
overpayment to Deposit Account No. 13-3848. A duplicate copy of this sheet is enclosed.
- d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card  
information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

**NOTE:** Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:



00157

PATENT TRADEMARK OFFICE

SIGNATURE

James R. Franks

NAME

42,552

REGISTRATION NUMBER

09/856545

JC18 Rec'd PCT/PTO 2 2 MAY 2001

PATENT APPLICATION  
Mo-6342  
WW-5522

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

APPLICATION OF )  
HARTWIG SCHLESIGER ) PCT/EP99/08779  
SERIAL NUMBER: TO BE ASSIGNED )  
FILED: HEREWITH )  
TITLE: METHOD FOR THE PRODUCTION )  
OF LOW-VISCOUS WATER- )  
SOLUBLE CELLULOSE ETHERS )

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents  
Washington, D.C. 20231  
Sir:

This preliminary amendment is being filed concurrently with the subject patent application. Upon granting a Serial Number and filing date, please amend the subject patent application as follows:

"Express Mail" mailing label number ET146895396US  
Date of Deposit May 22, 2001

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Assistant Commissioner of Patents and Trademarks, Washington, D.C. 20231

Donna J. Veatch

(Name of person mailing paper or fee)

  
(Signature of person mailing paper or fee)

### **IN THE SPECIFICATION:**

Please amend the specification as follows:

Please replace the title at the top of page 1 with the following

**--METHOD FOR THE PRODUCTION OF LOW-VISCOUS WATER-SOLUBLE  
CELLULOSE ETHERS--**

Please insert the following between lines 1 and 4 on page 1 of the  
specification.

**--CROSS REFERENCE TO RELATED PATENT APPLICATIONS**

The present patent application claims the right of priority under 35 U.S.C.  
119 and 35 U.S.C. 365 of International Application No. PCT/EP99/08779, filed 15  
November 1999, which was published in German as International Patent  
Publication No. WO 00/32636 on 8 June 2000, which is entitled to the right of  
priority of German Patent Application No. 198 54 770.6, filed 27 November  
1998.--

### **IN THE ABSTRACT**

**--METHOD FOR THE PRODUCTION OF LOW-VISCOUS WATER-SOLUBLE  
CELLULOSE ETHERS**

**ABSTRACT OF THE DISCLOSURE**

A process for preparing low-viscosity water-soluble cellulose ethers by the  
oxidative decomposition of higher-viscosity cellulose ethers with hydrogen  
peroxide is described. The process involves: (a) forming, under conditions of  
intensive mixing and at temperatures of 65 - 125°C, a mixture of, (i) one or more  
higher-viscosity cellulose ethers, and (ii) an aqueous solution of hydrogen  
peroxide, the proportions of the mixture being selected in such a way that the  
hydrogen peroxide content is 0.1 - 10 wt.% in relation to the dry cellulose ether,  
the solids content of the mixture is at least 25 wt.% in relation to the total weight  
of the mixture; and (b) agitating continuously the mixture of step (a) at  
temperatures of 65 - 125°C until at least approximately 90% of the hydrogen  
peroxide has been spent.--

A separate abstract page is included herewith.

**IN THE CLAIMS:**

Please add the following Claims 10, 11, 12 and 13:

--10. The process of Claim 1 wherein 0.2 to 2.5 wt. % hydrogen peroxide, in relation to the dry cellulose ether, is used.

11. The process of Claim 1 wherein 0.5 to 1.8 wt. % hydrogen peroxide, in relation to the dry cellulose ether, is used.

12. The process of Claim 1 wherein a higher-molecular cellulose ether with a solid content of 40 to 55 wt. %, in relation to the total quantity of cellulose ether and solvent, is used.

13. The process of Claim 6 wherein the pH value of the mixture is set at 6 to 7.--

Please replace Claim 1 with the following:

1. (Once Amended, Clean) A process for the production of low-viscosity water-soluble cellulose ethers by oxidative decomposition of higher-viscosity cellulose ethers with hydrogen peroxide, comprising:

- (a) forming, under conditions of intensive mixing and at temperatures of 65 - 125°C, a mixture comprising, (i) higher-viscosity cellulose ethers, and (ii) an aqueous solution of hydrogen peroxide, the proportions of the mixture being selected in such a way that the hydrogen peroxide content is 0.1 - 10 wt.% in relation to the dry cellulose ether, the solid content of the mixture is no lower than 25 wt.% in relation to the total quantity of the mixture; and
- (b) agitating continuously the mixture of step (a) at temperatures of 65 - 125°C until approximately at least 90% of the hydrogen peroxide has been spent.

Please replace Claim 2 with the following.

2. (Once Amended, Clean) The process of Claim 1 wherein mixing with the aqueous hydrogen peroxide solution is carried out step-by-step.

Please replace Claim 3 with the following.

3. (Once Amended, Clean) The process of Claim 1 wherein the mixture is agitated continuously at temperatures of 75 - 100°C.

Please replace Claim 4 with the following.

4. (Once Amended, Clean) The process of Claim 1 wherein 0.1 to 10

wt.% hydrogen peroxide in relation to the dry cellulose ether is used.

Please replace Claim 5 with the following.

5. (Once Amended, Clean) The process of Claim 1 wherein a higher-molecular cellulose ether with a solid content of 35 - 80 wt.%, in relation to the total quantity of cellulose ether and solvent, is used.

Please replace Claim 6 with the following.

6. (Once Amended, Clean) The process of Claim 1 wherein before, during or preferably after the decomposition reaction, the pH value of the mixture is set at more than 4.5, by mixing it with an aqueous solution which has a pH of 5 to 12 and optionally contains, in solution, the hydrogen peroxide required for the decomposition reaction.

Please replace Claim 7 with the following.

7. (Once Amended, Clean) The process of Claim 1 wherein the water soluble cellulose ether is selected from carboxymethyl cellulose, hydrophobically modified carboxymethyl cellulose, hydroxyethyl carboxymethyl cellulose, sulfoethyl cellulose, hydrophobically modified sulfoethyl cellulose, hydroxyethyl sulfoethyl cellulose, hydrophobically modified hydroxyethyl sulfoethyl cellulose, hydroxyethyl cellulose, hydrophobically modified hydroxyethyl cellulose, methyl cellulose, methylhydroxyethyl cellulose, methylhydroxyethyl sulfoethyl cellulose, hydrophobically modified methylhydroxyethyl cellulose, methylhydroxypropyl cellulose, hydroxypropyl cellulose and mixtures thereof.

Please replace Claim 8 with the following.

8. (Once Amended, Clean) The process of Claim 1 wherein the water-soluble cellulose ether is selected from methylcellulose, methyl hydroxyethyl cellulose, hydrophobically modified methyl hydroxyethyl cellulose, methyl hydroxypropyl cellulose, hydroxypropyl cellulose, mixtures thereof, and water-wet filter cakes of these cellulose ethers, as obtained after washing and separation.

Please replace Claim 9 with the following.

9. (Once Amended, Clean) The process of Claim 1 wherein the water soluble cellulose ether is selected from methyl hydroxyethyl cellulose, methyl hydroxypropyl cellulose, and water-wet filter cakes of these cellulose ethers, as obtained after washing and separation.

### REMARKS

Claims in the case are 1-13, upon entry of the present amendment.  
Claims 10-13 have been added, and Claims 1-9 have been amended herein.

Claims 1-9 of the above-identified patent application have been amended as to form, for example, by introducing indefinite and definite articles, replacing "characterized in that" with --wherein--, introducing gerunds into process Claim 1, and converting multi-dependent claims to dependent claims. Basis for added Claims 10 and 11 is found in original Claim 4, and at page 4, lines 23-25 of the specification. Basis for added Claim 12 is found in original Claim 5, and at page 4, lines 20-21 of the specification. Basis for added Claim 13 is found in original Claim 6, and at page 5, lines 1-10 of the specification.

The title of the patent application has been amended to correspond with the related PCT International Patent Publication No. WO 00/32636. Page 1 of the application has been amended herein to introduce cross reference information. The cross reference information is presented in accordance with 37 C.F.R. 1.78(a)(2) (Federal Register / Vol. 65, No. 183 / Wednesday, September 20, 2000; Changes to Implement Eighteen-Month Publication of Patent Applications; Final Rule). An abstract of the patent application is included herewith on a separate page.

The amendments presented herein do not represent the entry of new matter into the application. Applicant respectfully request entry of this amendment.

Respectfully submitted,

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Mo6342

## VERSIONS WITH MARKINGS TO SHOW CHANGES MADE

### IN THE SPECIFICATION: (Marked-Up)

The following is a version of the title at the top of page 1, with markings to show changes made thereto in the present Preliminary Amendment.

[Process for the production of low-viscosity water-soluble cellulose ethers.]

### METHOD FOR THE PRODUCTION OF LOW-VISCOUS WATER-SOLUBLE CELLULOSE ETHERS

### IN THE CLAIMS: (Marked-Up)

The following are versions of the amended claims with markings to show changes made thereto in the present Preliminary Amendment.

1. (Once Amended, Marked-Up) A process [Process] for the production of low-viscosity water-soluble cellulose ethers by oxidative decomposition of higher-viscosity cellulose ethers with hydrogen peroxide, [characterised in that,] comprising:

- (a) forming, under conditions of intensive mixing and at temperatures of 65 - 125°C, a mixture comprising, [the] (i) higher-viscosity cellulose ethers, [are intensively mixed with] and (ii) an aqueous solution of hydrogen peroxide [at temperatures of 65 - 125°C], the proportions of the mixture being selected in such a way that the hydrogen peroxide content is 0.1 - 10 wt.% in relation to the dry cellulose ether, the solid content of the mixture is no lower than 25 wt.% in relation to the total quantity of the mixture; and
- (b) agitating continuously the mixture of step (a) [is then agitated continuously] at temperatures of 65 - 125°C until approximately at least 90% [at least] of the hydrogen peroxide has been spent.

2. (Once Amended, Marked-Up) The process [Process according to claim] of Claim 1, characterised in that wherein mixing with the aqueous hydrogen peroxide solution is carried out step-by-step.

3. (Once Amended, Marked-Up) The process [Process according to claim] of Claim 1 [or 2, characterised in that] wherein the mixture is agitated continuously at temperatures of 75 - 100°C.



4. (Once Amended, Marked-Up) The process [Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims] of Claim 1 [to 3, characterised in that] wherein 0.1 to 10 wt.% hydrogen peroxide in relation to the dry cellulose ether[, preferably 0.2 to 2.5 wt.%, in particular 0.5 to 1.8 wt.% are] is used.

5. (Once Amended, Marked-Up) The process [Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims] of Claim 1 [to 4, characterised in that] wherein a higher-molecular cellulose ether with a solid content of 35 - 80 wt.%, [preferably 40 - 55 wt.%,] in relation to the total quantity of cellulose ether and solvent, is used.

6. (Once Amended, Marked-Up) The process [Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims] of Claim 1 [to 5, characterised in that] wherein before, during or preferably after the decomposition reaction, the pH value of the mixture is set at more than 4.5, [preferably 6 to 7,] by mixing it with an aqueous solution which has a pH of 5 to 12 and optionally contains, in solution, the hydrogen peroxide required for the decomposition reaction.

7. (Once Amended, Marked-Up) The process [Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims] of Claim 1 [to 6, characterised in that] wherein the water soluble cellulose ether is selected from carboxymethyl cellulose, hydrophobically modified carboxymethyl cellulose, hydroxyethyl carboxymethyl cellulose, sulfoethyl cellulose, hydrophobically modified sulfoethyl cellulose, hydroxyethyl sulfoethyl cellulose, hydrophobically modified hydroxyethyl sulfoethyl cellulose, hydroxyethyl cellulose, hydrophobically modified hydroxyethyl cellulose, methyl cellulose, methylhydroxyethyl cellulose, methylhydroxyethyl sulfoethyl cellulose, hydrophobically modified methylhydroxyethyl cellulose, methylhydroxypropyl cellulose, hydroxypropyl cellulose [or] and mixtures thereof.

8. (Once Amended, Marked-Up) The process [Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims] of Claim 1 [to 7, characterised in that] wherein the water-soluble cellulose ether is selected from methylcellulose, methyl hydroxyethyl cellulose, hydrophobically modified methyl hydroxyethyl cellulose, methyl hydroxypropyl cellulose,

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hydroxypropyl cellulose, [or] mixtures thereof, and water-wet filter cakes of these cellulose ethers, as obtained after washing and separation[, are used].

9. (Once Amended, Marked-Up) The process [Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims] of Claim 1 [to 7, characterised in that] wherein the water soluble cellulose ether is selected from methyl hydroxyethyl cellulose, [or] methyl hydroxypropyl cellulose, and water-wet filter cakes of these cellulose ethers, as obtained after washing and separation[, are used].

10. (Added) The process of Claim 1 wherein 0.2 to 2.5 wt. % hydrogen peroxide, in relation to the dry cellulose ether, is used.

11. (Added) The process of Claim 1 wherein 0.5 to 1.8 wt. % hydrogen peroxide, in relation to the dry cellulose ether, is used.

12. (Added) The process of Claim 1 wherein a higher-molecular cellulose ether with a solid content of 40 to 55 wt. %, in relation to the total quantity of cellulose ether and solvent, is used.

13. (Added) The process of Claim 6 wherein the pH value of the mixture is set at 6 to 7.

METHOD FOR THE PRODUCTION OF LOW-VISCOUS WATER-SOLUBLE  
CELLULOSE ETHERS

ABSTRACT OF THE DISCLOSURE

A process for preparing low-viscosity water-soluble cellulose ethers by the oxidative decomposition of higher-viscosity cellulose ethers with hydrogen peroxide is described. The process involves: (a) forming, under conditions of intensive mixing and at temperatures of 65 - 125°C, a mixture of, (i) one or more higher-viscosity cellulose ethers, and (ii) an aqueous solution of hydrogen peroxide, the proportions of the mixture being selected in such a way that the hydrogen peroxide content is 0.1 - 10 wt.% in relation to the dry cellulose ether, the solids content of the mixture is at least 25 wt.% in relation to the total weight of the mixture; and (b) agitating continuously the mixture of step (a) at temperatures of 65 - 125°C until at least approximately 90% of the hydrogen peroxide has been spent.

Process for the production of low-viscosity water-soluble cellulose ethers.

- 5 The technological properties of cellulose ethers are highly dependent on the viscosity of their solutions. Although primarily medium-viscosity cellulose ethers, i.e. those with average molecular weight are processed, high- and low-viscosity cellulose ethers have nevertheless also achieved importance.

- 10 Low-viscosity cellulose ethers, which also have a low molecular weight in comparison with medium- and high-viscosity cellulose ethers, can in principle be manufactured in two different ways. Either a low-molecular alkali cellulose is taken as the basis and etherified, or a finished cellulose ether is broken down to the desired molecular weight.

- 15 Using a low-molecular alkali cellulose as the basis and producing a cellulose ether by etherification makes the subsequent cleaning process more difficult. The cellulose ether contains a considerable number of short-chain components, which are swollen greatly or washed out by the washing media.

- 20 The second possible method mentioned, of breaking down higher-molecular cellulose ethers into low-molecular, low-viscosity cellulose ethers can be achieved by the action of oxidising agents, for example hypochlorite or hydrogen peroxide.

The oxidative decomposition of high-viscosity cellulose ethers can be carried out after the cleaning process. This avoids washing losses and difficulties during the washing process.

The specifications listed below give a summary of the processes currently used to break down high-viscosity cellulose ethers after etherification and washing:

Date of deposit \_\_\_\_\_  
 "Express Mail" mailing label number EP146893396US  
May 22, 2001

I hereby certify that this paper or leaf is being deposited with the United States Postal Service "Express Mail" Post Office to Addressee" service under 37 CFR 1.10 On the date indicated above and is addressed to the Assistant Commissioner of Patents and Trademarks, Washington, D.C. 20231

Domina J. Veatch

(Name of person mailing paper or leaf)

Signature of person mailing paper or leaf

DE 2 016 203 from The Dow Chemical Co. claims a process for reducing the viscosity of cellulose ethers with hydrogen peroxide. For this process a substantially dry, free-flowing cellulose ether with a water content of less than 5 wt.% is mixed with a 10 to 50% hydrogen peroxide solution and the mixture obtained is heated to 50 to 150°C.

DE 1 543 116 from Kalle AG claims a process for the production of low-viscosity cellulose ethers by oxidative decomposition of higher-viscosity cellulose ethers with hydrogen peroxide. This process is characterised in that, a higher-viscosity cellulose ether is mixed with an aqueous solution of hydrogen peroxide, the water content of the mixture not exceeding 75 wt.% in relation to the total quantity. The mixture is then dried at temperatures of 100°C to 250°C until the hydrogen peroxide is spent. Here, the loss of moisture and of hydrogen peroxide run virtually in parallel with the reduction in viscosity.

These processes have in common, that a low-viscosity cellulose ether in dry powder or granule form results directly from the decomposition reaction. Either drying is carried out before the decomposition reaction and the process is carried out with low moisture contents, or the process begins with a wet product and ends with low moisture contents. The loss of viscosity then runs virtually in parallel with the loss of moisture.

The object was to provide a process which allows the viscosity to be set immediately after washing of the cellulose ether in such a way that the subsequent drying, shaping (grinding, granulation) and mixing are not affected and that the decomposition reaction is not affected by the subsequent process steps drying, shaping (grinding, granulation) and mixing.

This object was achieved in that a higher-viscosity water-soluble cellulose ether as obtained after washing, was mixed with an aqueous solution of hydrogen peroxide,

the dry content of the mixture not exceeding 25 wt.% in relation to the total quantity. The mixture is then continuously agitated at temperatures of 65 - 125°C, preferably 75 - 100°C, until the hydrogen peroxide is spent, and then dried.

5 By this process a low-viscosity water-soluble cellulose ether is obtained. Surprisingly the subsequent process steps for the production of cellulose ethers ready-for-sale, such as drying, shaping (grinding, granulation) and mixing are not affected by the decomposition reaction. The degree of moisture and grinding can be set independently of the reduction in viscosity.

10 Low-viscosity cellulose ethers are deemed here to be cellulose ethers of which 2% aqueous solutions have viscosities of 2 to 400, in particular 2 to 100 mPa/s (Haake Rotovisko) at 20°C and a shear rate of  $2.55 \text{ s}^{-1}$ . A higher-viscosity cellulose ether is deemed here to be a cellulose ether of which 2% aqueous solutions have a viscosity  
15 of 100 to 100,000, preferably 400 to 20,000 mPa/s at 20°C and at a shear rate of  $2.55 \text{ s}^{-1}$ . Here the viscosity reduction in the end product as compared with the raw material, brought about by the process according to the invention, preferably amounts to at least 50%, in particular 70%, and more particularly 98%.

20 Ionic or non-ionic cellulose ethers may be used as raw materials, such as preferably carboxymethyl cellulose, hydrophobically modified carboxymethyl cellulose, hydroxyethyl carboxymethyl cellulose, sulfoethyl cellulose, hydrophobically modified sulfoethyl cellulose, hydroxyethyl sulfoethyl cellulose, hydrophobically modified hydroxyethyl sulfoethyl cellulose, hydroxyethyl cellulose, hydrophobically  
25 modified hydroxyethyl cellulose, methyl cellulose, methyl hydroxyethyl cellulose, methyl hydroxyethyl sulfoethyl cellulose, hydrophobically modified methyl hydroxyethyl cellulose, methyl hydroxypropyl cellulose, hydroxypropyl cellulose and mixtures or derivatives thereof. Preferred raw materials are in particular methyl hydroxyethyl cellulose or methyl hydroxypropyl cellulose. It is advantageous to use

water-wet filter cakes of these cellulose ethers, as obtained after washing and separation.

5 The process can easily be incorporated into the normal method of production of a cellulose ether. The higher-viscosity raw material is centrifuged after washing to a dry content of 25 to 80 wt.% in relation to the total weight.

10 Then an aqueous solution of hydrogen peroxide at temperatures of 65 to 125°C, is incorporated intensively, optionally step-by-step, the proportions of the mixture being selected in such a way that the hydrogen peroxide content is 0.1 to 10 wt.% in relation to the dry matter and the dry content of the mixture does not fall below 25 wt.% in relation to the total quantity. The mixture is then agitated continuously at temperatures of 65-125°C, preferably temperatures of 75-100°C until approximately 90% at least, preferably 95% at least of the hydrogen peroxide used is spent. It is  
15 preferred more particularly that the mixture be agitated continuously until the hydrogen peroxide has been fully spent. The other process steps such as the addition of additives, crosslinking with di-aldehydes, compression, drying and grinding can then be carried out in the usual way.

20 A higher-molecular cellulose ether with a dry content of 35 to 80 wt.%, in particular 40 to 55 wt.% in relation to the total quantity, is preferably used in the process.

0.1 to 10 wt.% hydrogen peroxide in relation to the dry cellulose ether, preferably 0.2 to 2.5 wt.%, in particular 0.5 to 1.8 wt.% in relation to the dry cellulose ether is  
25 used to break down the higher-viscosity cellulose ethers.

Depending on the raw material, products with 2 wt.% aqueous solutions having acid pH values of 3 to 5 result from the decomposition reaction. It has proved useful here, before, during or after the decomposition reaction, but in any case before any further  
30 processing step such as drying or shaping, to set the pH value of the product.

Particularly good results are achieved if the pH value is set after the decomposition reaction. The pH value is set using aqueous solutions of salts, which have a pH value of 5 to 12 and optionally contain all or part of the required hydrogen peroxide. These solutions are added to the reaction mixture in such quantities, that the pH value of the mixture after addition is set at more than 4.5, preferably 6 to 7. Aqueous solutions of sodium dihydrogen phosphate, sodium hydrogen phosphate, sodium phosphate, sodium carbonate and sodium hydrogen carbonate or aqueous solutions of mixtures of these salts are advantageously used. Alkali salts of weak acids such as those of citric acid or succinic acid in the form of aqueous buffer solutions can also be used.

An advantage of the claimed process is that the actual reduction in molecular weight is completely separated from subsequent drying. This has the advantage that any type of drying equipment, with varying residence time requirements of the cellulose ether particles to be dried, can be used without affecting the decomposition reaction. Furthermore, only one piece of equipment, the mixer in which the decomposition reaction is to be carried out, is affected by the corrosive properties of the hydrogen peroxide incorporated. In particular, it is possible to incorporate additives and modifiers after the decomposition reaction, but before drying, into the solvent-wet (e.g. water-wet) cellulose ether. Here, the group of dialdehydes (e.g. glyoxal) is mentioned in particular. These compounds are used to produce solvent-inhibited cellulose ethers. They cannot be used together with the hydrogen peroxide required for the decomposition reaction because of their sensitivity to oxidation. It is also possible to mix in oligomeric or polymeric oxidation-sensitive substances (e.g. polysaccharides, polysaccharide ethers, polyvinyl alcohol, polyester, polyamide) after the decomposition reaction and before drying.

The following examples explain the present invention further.



**Examples 1-4**

5 kg quantities of methylhydroxyethyl cellulose with a methoxy group content of 24.2-30.5 % and a hydroxyethoxy group content of 7.5-14.8 % and a moisture content of 50 - 53 wt.%, in relation to the total quantity and with a viscosity as given in the following table, measured on 2 wt.% aqueous solutions at 20°C and at a shear rate of 2.55 s<sup>-1</sup> (Haake Rotovisko), were sprayed with 800 ml aqueous hydrogen peroxide solution. The mixture thus obtained was agitated continuously for 6 hours at 75°C and then dried.

The quantities of hydrogen peroxide used, the initial viscosity and the final viscosity are shown in the table. The quantities stated relate to the dry methylhydroxyethyl cellulose.

Number	Initial viscosity (mPa/s)	H <sub>2</sub> O <sub>2</sub> added wt.%	Final viscosity (mPa/s)
1	7,100	0.5	134
2	7,100	1.0	70
3	7,100	1.5	21
4	400	1.5	6

**Examples 5-7**

5 kg quantities of methylhydroxyethyl cellulose with a methoxy group content of 21.4 - 26.1 % and a hydroxyethoxy group content of 5.9 - 9.8 % and a moisture content of 52 wt.% in relation to the total quantity and with a viscosity as given in the following table, measured on a 2 wt.% aqueous solution at 20°C and a shear rate of 2.55 s<sup>-1</sup> (Haake Rotovisko), were sprayed with 500 ml aqueous hydrogen peroxide solution. The quantity of hydrogen peroxide used was 1 wt.% in relation to the dry

methylhydroxyethyl cellulose. The mixture thus obtained was agitated continuously until the hydrogen peroxide had been spent and then dried.

The reaction temperatures, reaction times, initial and final viscosities in each case are shown in the table.

Number.	Initial viscosity (mPa/s)	Final viscosity (mPa/s)	Reaction temperature (°C)	Reaction time (h)
5	6,000	40	75	6
6	6,000	34	85	5
7	6,000	36	95	3

#### **Examples 8-10**

5 kg quantities of methylhydroxyethyl cellulose with a methoxy group content of 21.4 - 26.1 %, a hydroxyethoxy group content of 5.9 - 9.8 % and a moisture content of 52 wt.% in relation to the total quantity and with a viscosity as given in the following table, measured on a 2% aqueous solution at 20°C and a shear rate of 2.55 s<sup>-1</sup> (Haake Rotovisko), were sprayed with 500 ml aqueous hydrogen peroxide solution. The mixture thus obtained was agitated continuously for 3 hours at 95°C until the hydrogen peroxide was spent and then sprayed with 250 ml of an aqueous solution of sodium hydrogen phosphate and sodium carbonate and mixed for a further 60 minutes. It was then dried.

The initial and final viscosities in each case, the quantities of sodium hydrogen phosphate and sodium carbonate used, the pH values of 2 wt.% solutions of the products and the quantities of hydrogen peroxide used are shown in the table. The quantities given relate to the dry methyl hydroxyethyl cellulose.

Number	Initial viscosity (mPa/s)	Final viscosity (mPa/s)	Sodium hydrogen phosphate (wt.-%)	Sodium carbonate (wt.-%)	pH-values of 2 wt.-% aqueous solutions	H <sub>2</sub> O <sub>2</sub> - added (wt.-%)
8	6,000	36	0.25	0.2	5.6	1.0
9	6,000	29	0.25	0.3	5.9	1.0
10	6,000	19	0.25	0.5	5.0	1.5

### Examples 11-12

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5 kg quantities of methylhydroxyethyl cellulose with a methoxy group content of 24.2 - 30.5 % and a hydroxyethoxy group content of 7.5 - 14.8 % and a moisture content of 50 - 53 wt.% in relation to the total quantity and with a viscosity as given in the following table, measured on 2 wt.-% aqueous solutions at 20° C, and at a shear rate of 2.55 s<sup>-1</sup> (Haake Rotovisko), were sprayed with 800 ml aqueous hydrogen peroxide solution. The quantity of hydrogen peroxide used was 1.5 wt.% in relation to the dry methylhydroxyethyl cellulose. An additional 0.5 wt.% (in relation to the dry methylhydroxyethyl cellulose) sodium citrate was added to the hydrogen peroxide solution. The mixture thus obtained was agitated continuously for 5 hours at 90°C until the hydrogen peroxide was spent and then dried.

The initial and final viscosities in each case and the pH values of 2 wt.-% solutions of the products are shown in the table.



**Patent claims**

1. Process for the production of low-viscosity water-soluble cellulose ethers by oxidative decomposition of higher-viscosity cellulose ethers with hydrogen peroxide, characterised in that, the higher-viscosity cellulose ethers are intensively mixed with an aqueous solution of hydrogen peroxide at temperatures of 65 - 125°C, the proportions of the mixture being selected in such a way that the hydrogen peroxide content is 0.1 - 10 wt.% in relation to the dry cellulose ether, the solid content of the mixture is no lower than 25 wt.% in relation to the total quantity of the mixture and the mixture is then agitated continuously at temperatures of 65 - 125°C until approximately 90% at least of the hydrogen peroxide has been spent.
2. Process according to claim 1, characterised in that mixing with the aqueous hydrogen peroxide solution is carried out step-by-step.
3. Process according to claim 1 or 2, characterised in that the mixture is agitated continuously at temperatures of 75 - 100°C.
4. Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims 1 to 3, characterised in that 0.1 to 10 wt.% hydrogen peroxide in relation to the dry cellulose ether, preferably 0.2 to 2.5 wt.%, in particular 0.5 to 1.8 wt.% are used.
5. Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims 1 to 4, characterised in that a higher-molecular cellulose ether with a solid content of 35 - 80 wt.%, preferably 40 - 55 wt.%, in relation to the total quantity of cellulose ether and solvent, is used.

6. Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims 1 to 5, characterised in that before, during or preferably after the decomposition reaction, the pH value of the mixture is set at more than 4.5, preferably 6 to 7, by mixing it with an aqueous solution which has a pH of 5 to 12 and optionally contains, in solution, the hydrogen peroxide required for the decomposition reaction.
7. Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims 1 to 6, characterised in that the water soluble cellulose ether is carboxymethyl cellulose, hydrophobically modified carboxymethyl cellulose, hydroxyethyl carboxymethyl cellulose, sulfoethyl cellulose, hydrophobically modified sulfoethyl cellulose, hydroxyethyl sulfoethyl cellulose, hydrophobically modified hydroxyethyl sulfoethyl cellulose, hydroxyethyl cellulose, hydrophobically modified hydroxyethyl cellulose, methyl cellulose, methylhydroxyethyl cellulose, methylhydroxyethyl sulfoethyl cellulose, hydrophobically modified methylhydroxyethyl cellulose, methylhydroxypropyl cellulose, hydroxypropyl cellulose or mixtures thereof.
8. Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims 1 to 7, characterised in that the water-soluble cellulose ether is methylcellulose, methyl hydroxyethyl cellulose, hydrophobically modified methyl hydroxyethyl cellulose, methyl hydroxypropyl cellulose, hydroxypropyl cellulose or mixtures thereof and water-wet filter cakes of these cellulose ethers, as obtained after washing and separation, are used.
9. Process for the production of low-viscosity water-soluble cellulose ethers according to any one of claims 1 to 7, characterised in that the water soluble cellulose ether is methyl hydroxyethyl cellulose or methyl hydroxypropyl

cellulose and water-wet filter cakes of the cellulose ethers, as obtained after washing and separation, are used.

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As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

**METHOD FOR THE PRODUCTION OF LOW-VISCOUS WATER-SOLUBLE CELLULOSE ETHERS**

the specification of which is attached hereto,

or was filed on **November 15, 1999**

as a PCT Application Serial No. **PCT/EP99/08779**

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

**198 54 770.6**  
(Number)

**Germany**  
(Country)

**November 27, 1998**  
(Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



**POWER OF ATTORNEY:** As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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